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DOUBLE DOPED RAP FLUORIDES.(U)
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DOUBLE DOPED RAP FLUORIDES

M. Robinson

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3011 Malibu Canyon Road
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Single crystals of calcium fluoride and strontium fluoride double doped with Na^{+} and Ce^{3+} were czochralski grown using RAP (HF) to maximize anion purity.

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SECTION 1

INTRODUCTION

The objective of this program is the production of single crystals of calcium fluoride (CaF_2) and strontium fluoride (SrF_2), both double doped with Na^+ and Ce^{3+}. The crystals were grown by the Czochralski method utilizing hydrogen fluoride (HF) RAP to maximize anion purity.

SECTION 2

EQUIPMENT AND MATERIALS

A. EQUIPMENT

1. Astro Industries graphite resistance furnace with stainless steel shell

B. MATERIALS

1. HF - Matheson 99.9%
2. Helium (He) - Airco 99.995%
3. CaF_2 EM Labs 99.999%
4. SrF_2 EM Labs 99.999%
5. CeF_3 Research Chemicals 99.99%
6. NaF MCB Reagent grade
7. Vitreous carbon crucibles from Beckwith Corporation

SECTION 3

EXPERIMENTAL

A. MELT PREPARATION AND DISCUSSION

The host material and its dopants, all in powder form, were contained in a 50-mm diameter vitreous carbon crucible and situated at the top of the furnace hot zone. The furnace was evacuated with a roughing pump and the temperature was increased slowly to ∿600°C for outgassing. The furnace was then backfilled with He and a flow of 1 LPM was initiated. The temperature was then rasied slowly to approximately 100°C higher than the pulling temperature (Tables 1 and 2). During this slow temperature rise the dopants diffuse into the host lattice to form a solid solution. This decreases the volatility of NaF and its reactivity to HF. At this point the melt is ready for an HF atmosphere and a minimum loss of NaF is ensured. The preliminary He melting step is important, because previous work at HRL has shown that losses from the crucible were high when pure NaF was melted in an HF atmosphere. HF reacts to form $NaHF_2$, which is readily vapor-transported to the cold portions of the furnace. However, when NaF can first be dissolved in the host in the absence of RAP, as described above, subsequent meltings in HF do not promote sodium loss.

B. CRYSTAL GROWTH

After the first He melting the atmosphere is adjusted by flow-rate manipulation to give 90% He and 10% HF. The melt is allowed to see this atmosphere for 4 hours before the start of crystal growth.

Growth is spontaneously nucleated on a 3 mm platinum rod and a steady state growth rate of 9.5 mm-hr^{-1} is enforced. At the end of the run the crystal is withdrawn from the melt rapidly and the furnace is allowed to cool to room temperature.

Table 1. Crystal Growth Conditions and Results for Double Doped CaF_2

Processing Data: ATMOSPHERE - 90% He - 10% HF
PULLING TEMPERATURE ∿ 1450°C
PULL RATE - 9.5 mm-hr^{-1}
ROTATION RATE - 29 R.P.M.
COOL DOWN - 2 Hrs.

	Mole % CaF_2	CeF_3	NaF	Starting Weight (g)	Crystal Weight (g)	Residue (g)	Wt. Loss (g)
Prep 1*	99.8835	0.0104	0.1060				
1478	-	-	-	170.76	7.20	-	-
Prep 2	99.8845	0.0110	0.1046				
1978	-	-	-	170.90	13.2	157.45	0.18
1478	-	-	-	157.45	5.17	151.93	0.35
Prep 3	99.8722	0.022	0.1059				
11678	-	-	-	175.28	4.16	170.87	0.25
11978	-	-	-	170.87	14.64	156.16	0.32

*Prep. 1 consists of a boule grown from CaF_2 chunks instead of powder, with ends polished for spectroscopic examination. This is a complimentary crystal.

Table 2. Crystal Growth Conditions and Results for Double Doped SrF_2

Processing Data: ATMOSPHERE - 90% He - 10% HF
PULLING TEMPERATURE ∿ 1500°C
PULL RATE - 9.5 mm-hr^{-1}
ROTATION RATE - 29 R.P.M.
COOL DOWN - 2 Hrs.

	Mole % SrF_2	CeF_3	NaF	Starting Weight (g)	Crystal Weight (g)	Residue (g)	Wt. Loss (g)
Prep. 4	99.8882	0.0107	0.1012				
12078*	-	-	-	213.71	1.92	211.79	0
12378	-	-	-	211.79	19.25	192.24	0.30
12478	-	-	-	191.96	8.10	183.64	0.22
Prep. 5	99.8925	0.0063	0.1013				
2678**	-	-	-	243.00	20.74	221.91	0.35
2778	-	-	-	221.91	16.60	204.96	0.35
2978***	-	-	-	204.96	17.64	187.04	0.28

*Growth run 12078, was aborted due to equipment malfunction.

**For growth run 2678, crystal diameter was considered too irregular for delivery. However, the crystal was delivered and should be considered complimentary.

***Cooldown on crystal 2978 was as follows: Cooled to ∿ 300°C below melting point in 16 hours. Then, cooled to room temperature in 2 hours.